

METHOD AND APPARATUS FOR CONTINUOUS FLOW REDUCTION
OF MICROBIAL AND/OR ENZYMATIC ACTIVITY
IN A LIQUID PRODUCT USING CARBON DIOXIDE

5 This application is a continuation-in-part of U.S.
patent application Serial No. 09/314,945, filed May 20,
1999, and claims priority from U.S. provisional
application Serial No. 60/095,967 filed August 10,
1998.

10 FIELD OF THE INVENTION

This invention relates to a method and apparatus
for the processing of liquids to reduce microbial
and/or enzymatic activity therein and, more
particularly, to the use of carbon dioxide to achieve
15 reductions of microbial and/or enzymatic activity.

BACKGROUND OF THE INVENTION

There are many methods for improving the shelf
life of liquid products such as orange juice, apple
20 juice, milk, latex paints, peanut butter, soup, etc.

Commercially, thermal methods such as
pasteurization are the predominant methods used to
improve the shelf life of liquid foods. Ultra-high
pressure treatment is also used for liquid foods, but
25 less frequently.

In high pressure treatment facilities, fluids
containing microbial contamination are pressurized
hydrostatically to kill the majority of the bacteria.
In such systems, pressures are created which equal or
30 exceed 30,000 psia and commonly range from 60,000 to
120,000 psia. Such hydrostatic treatment, however, is
unsafe because of the very high pressures, is a lengthy
process, is batch rather than continuous, and is

expensive due to the high capital costs of the required equipment.

Other methods for shelf-life extension of liquids include nuclear irradiation, ultra-violet exposure and
5 application of microwaves. These treatments are expensive and not widely used commercially at present.

High pressure homogenization has been used to increase the shelf life of orange juice and other single-strength citrus juices as described in U.S.
10 Patent 5,232,726 to Clark et al. It is disclosed that a citrus juice being processed is subjected to a high pressure of about 15,000 psia, with the result being a significant reduction in biological activity in the juice.

15 Carbon dioxide has been used to inactivate enzymes in food and reduce microbial populations in fruit juices as described in U.S. Patent 5,393,547 to Balaban et al. Balaban et al. describe a method for inactivating enzymes in liquid food products wherein
20 the food is exposed to pressurized carbon dioxide which, in turn, produces a carbonic acid solution with a pH that is sufficiently low to irreversibly inactivate enzymes in the liquid food. The Balaban et al. method is indicated as being applicable to either
25 batch mode or continuous flow mode processing of food. Balaban et al. further indicate that supercritical carbon dioxide is introduced at a rate sufficient to allow enough thereof to dissolve in the food to inactivate the enzymes. After enzymatic inactivation,
30 the food flows to a section where pressure is reduced and the released carbon dioxide may be recycled for repeat usage.

U.S. Patent 5,704,276 to Osajima et al. describes a method for continuous deactivation of enzymes in liquid foodstuffs, using a supercritical form of carbon dioxide. Osajima et al. indicate that the density of the supercritical fluid is less than that of the liquid food and that the supercritical carbon dioxide is injected continuously into the liquid food and is separated therefrom in a later stage of the process. Osajima et al. also indicate that their process deodorizes the liquid food and removes volatile components.

Arreola et al. in "Effect of Supercritical Carbon Dioxide on Microbial Populations in Single Strength Orange Juice", Journal of Food Quality, Volume 14 (1991), pp. 275-284, describe the effect of supercritical carbon dioxide on microbial populations in orange juice. Using a batch process, Arreola et al. concluded that high pressure carbon dioxide treatment resulted in microbial reduction in single strength orange juice, even at low temperatures. Further, they conclude that a combination of high pressure, and shear forces to which the orange juice is subjected during depressurization and lower pH due to temporary formation of carbonic acid may have further inhibitory effects on the normal flora within orange juice. During the processing described in this paper, the minimum temperature utilized was 35°C.

It is an object of this invention to provide an improved method and apparatus for reducing microbial and/or enzymatic activity in liquid products.

It is a further object of this invention to

provide a method and apparatus for reducing microbial and/or enzymatic activity in liquid products using pressurized carbon dioxide, wherein the processing temperature to which the liquid is subjected does not deleteriously affect the liquid products.

It is yet another object of this invention to provide a continuous flow method and apparatus for reducing microbial and/or enzymatic activity in liquid products using pressurized carbon dioxide.

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SUMMARY OF THE INVENTION

A continuous method using a pressurized flow of carbon dioxide is described for the reduction of microorganisms present in the liquid product and/or the inactivation of one or more enzymes in a pressurized flow of the liquid product. In one embodiment, the pressure in the flow regions is maintained at a level which is sufficient to keep the carbon dioxide in dense phase, but at a temperature which does not freeze the liquid product. In another embodiment, gaseous carbon dioxide is injected directly into the liquid product, forming a mixture which is thereafter pressurized.

The pressurized mixture of the carbon dioxide and liquid flows through a reaction zone for a sufficient time to reduce harmful microorganisms and inactivate enzymes and then enters one or a plurality of expansion stages wherein the pressure of the mixture flow is decreased sufficiently to allow the separation of carbon dioxide from the liquid product. Heat is applied if necessary, to the extent necessary, in at least some of the expansion stages to prevent a cooling of the mixture flow to the freezing point of the liquid

product. If heat is applied, the temperature should preferably be controlled so that the liquid does not exceed a temperature at which deleterious effects are experienced. (Freezing and excessive high temperature
5 can have negative effects on the juice quality. Temperatures over 40°C begin to degrade the product.)

The present invention is contemplated for use with any fluid that may be transported through a conduit, including for example, beverage products such as juices
10 and milk, semi-liquid foods such as mayonnaise, salad dressings, soup and cottage cheese, and other fluids such as paint and sterile injectibles.

BRIEF DESCRIPTION OF THE DRAWING

15 Figure 1 is a schematic flow diagram of apparatus which performs one embodiment of the invention.

Figure 2 is a schematic flow diagram of apparatus which performs another embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

20 Referring to Figure 1, pressurized carbon dioxide is fed from carbon dioxide supply 10 through optional pressure regulator 12 to a pump 14 which increases the pressure of the carbon dioxide flow and then feeds it
25 through a check valve 16 to a juncture 18. The carbon dioxide is pressurized at pump 14 to prevent any boiling of the dense phase carbon dioxide during later stages of the process.

In similar fashion, liquid product is fed from a
30 liquid product feed tank 20 through a valve 22 to a pump 24. Pump 24 raises the feed pressure of the

liquid product to the same level as that of the dense phase carbon dioxide exiting from pump 14. The pressurized liquid product feed passes through check valve 26 to juncture 18 where it combines with the
5 pressurized flow of carbon dioxide. The mixture of the liquid product and carbon dioxide then passes to an in-line mixer 28 (optional) which essentially comprises a heavily baffled conduit that thoroughly mixes the carbon dioxide and liquid product streams. Of course,
10 other mixers may be employed which achieve a desired level of liquid product/carbon dioxide mixing. The liquid mixture exits from in-line mixer 28 and is further pressurized by the action of pump 30 to a process pressure.

15 Depending upon the specific liquid product feed, the process pressure will vary accordingly. It is preferred that the process pressure be within the range of 300 psia to 20,000 psia. If orange juice is being processed as a liquid food, a preferred range of
20 pressure is about 3000 psia to about 7000 psia.

Referring to Figure 2, carbon dioxide is fed from source 110 through optional pressure regulator 112. Pump 114 can pressurize the carbon dioxide to dense phase or liquid and convey it to juncture 118, or if
25 the carbon dioxide is gaseous then pump 114 can be omitted and the gas flows under its own pressure to juncture 118. Separately, liquid product is fed from liquid product feed tank 120 through valve 122. Preferably, a pump 124 helps convey the liquid product
30 to juncture 118 but need not pressurize the liquid product.

The liquid product and the carbon dioxide are

mixed together, in-line (for instance at juncture 118) or for instance with the aid of optional mixing device 128 (which could be at juncture 118) . If the carbon dioxide is liquid, an in-line mixer or equivalent
5 device can be used as described with respect to device 28 in Figure 1. If the carbon dioxide is gaseous, any device effective to feed the gas into the liquid product can be used, such as a sparger, in-line injector, sidestream injection, ultrasonic transducers,
10 or mixing with dry ice. Injection devices include membranes, sintered metal spargers, flexible diffusers, sidestream ejectors, venturi injectors, and equivalent ("Praso") valves. The gaseous carbon dioxide can be fed into the feed line through which the liquid product
15 passes, or into a holding tank (not shown) located at a point in the feed line between juncture 118 and pump 130. Then the mixture is pressurized at pump 130 to process pressure.

Once the liquid mixture however formed exits from
20 pump 30 or 130, it enters a reaction zone 32 that is of suitable size and length to provide sufficient contact (or residence) time for the carbon dioxide and liquid product to interact in a manner which reduces microorganisms and/or inactivates enzymes including
25 undesirable enzymes present in the liquid product. The selected residence time will depend on the liquid product to be processed and its flowrate, as well as the size and length of the reaction zone. It is preferred that the reaction zone residence time is in
30 the range of about 1.0 to about 15.0 minutes.

For example, for processing orange juice, at a flowrate of 500 ml/min in a reaction zone having a

length of about 100 feet and tubing size of about 0.56 inches (142 mm) inner diameter (I.D.), the preferred residence time is about 1.5 to 13.0 minutes, and more preferably about 3.0 minutes of residence time.

5 As the liquid mixture stream exits from reaction zone 32, it enters one or more interaction chambers 34 (optional) wherein high shear forces are applied which enable a rupture of microbial cell walls in the liquid mixture. Such action enables a further reduction of
10 the microbial populations in the liquid mixture. For example, a high shear interaction chamber can be used, one example of which suitable for inclusion in this process is manufactured by the Microfluidics International Corp., Newton, Massachusetts.
15 Homogenizers are also useful for this purpose.

 At this stage, the pressurized carbon dioxide/liquid product mixture must be depressurized in such a fashion as to avoid freezing the liquid product (due to the Joule-Thompson cooling effect of the
20 expansion of the carbon dioxide). If the pressure is lowered to ambient in one or two stages, application of supplemental heat may be required. If too much heat is added to the mixture, damage will occur to the liquid product, either in its flavor characteristics or its
25 composition. Also, important volatiles such as flavor components may be carried away. Accordingly, it has been found that substantial care must be taken during the depressurization action to maintain the liquid mixture within two boundaries. The lower boundary is
30 the freezing point of the liquid mixture and the upper boundary point is the maximum temperature to which the liquid product can be subjected, without damage to the

product.

In the case of orange juice, the maximum temperature is about 60°C and the minimum temperature is about 0°C. Accordingly, when choosing a pressure reduction scheme, a pressure/enthalpy chart for carbon dioxide is followed to determine the optimum pressure and heating temperature needed for plural pressure reduction stages, while keeping (in this example) the orange juice at a temperature between that which will injure its flavor and its freezing point. It has been determined that at least two stages of depressurization are preferred, but one or multiple stages are possible.

Returning to Figure 1, while one or more depressurization stages can be used, three are shown. The first depressurization stage includes a pressure control device 36, such as a back pressure regulator, followed by a heat exchanger 38. Assuming that the liquid product being processed is orange juice and that the process pressure within reaction zone 32 and (optional) interaction chamber 34 is about 5,000 psig, a first depressurization stage 35 reduces the pressure of the liquid mixture to approximately 500 psig and applies sufficient heat through heat exchanger 38 to maintain the liquid mixture at about 20°C.

A second optional depressurization stage 40 includes a pressure control device 42 and heat exchanger 44 which, in combination, reduce the pressure of the liquid mixture to about 250 psia and maintains its temperature at approximately 30°C. A final stage depressurizer 46 includes only a pressure control device 48 to reduce the pressure of the liquid mixture

to the point where the dense phase carbon dioxide will vaporize and may be separated from the liquid products while minimizing loss of important volatile components. In the embodiment shown in the figure, no heat
5 exchanger is required subsequent to pressure control device 48, however, one may be provided, if required, to maintain the liquid mixture within the required temperature range.

As the liquid mixture exits from pressure control
10 device 48, it enters a liquid product/carbon dioxide separator vessel 50 or other collection device at reduced pressure. There, the carbon dioxide vapor separates from the liquid product, is captured and (if desired) is passed through optional filter 52 and/or
15 optional flow meter 54 and is either vented to atmosphere or is passed through a pressurization stage (not shown) for recycling back to carbon dioxide supply 10. The liquid product pool 56 may then be drained through valve 58 for subsequent processing and/or use.
20 There may be included a stage (not shown) for reducing residual dissolved carbon dioxide to desired levels, e.g. from on the order of 1200 ppm down to 300-400 ppm or less.

It is to be understood, that the continuous
25 process method shown in the figure is made practical by the one or more, preferably multiple, depressurization stages which enable the liquid mixture to be maintained within the aforementioned temperature boundaries. As a result, a continuous process for reduction of microbial
30 and/or enzymatic activity is achieved while overcoming the principal problem of the prior art, i.e., batch processing which is an uneconomic and undesired

processing procedure in a commercial environment.

If the carbon dioxide gas is to be recycled, it may be passed through a coalescing filter to remove droplets of the processed liquid product. Thereafter, 5 the gas is recondensed, or compressed, to the liquid state by passage through a condensing heat exchanger or compressor. Further, to assure removal of the dissolved carbon dioxide in the processed liquid product, a liquid product/carbon dioxide separator 10 downstream from separator tank 50 may include means for dissolved gas removal.

The resultant gas, remaining after processing, may carry additional valuable aromas and/or flavors. To recover or remove such aromas or flavors, a method such 15 as condensation or absorption may be utilized.

It should be understood that the foregoing description is only illustrative of the invention. Various alternatives and modifications can be devised by those skilled in the art without departing from the 20 invention.